VII. COSY - 2d Homonuclear Correlation

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A. Discussion

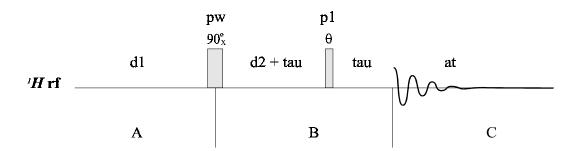
- Absolute value mode COSY often provides sufficient homonuclear correlation data in a very efficient, robust experiment.
- $J_{HH} \ge 4$ Hz can typically be observed, but this is dependent on sw and ni

$$digital\ resolution in F2 = \frac{sw}{np} \qquad digital\ resolution in F1 = \frac{sw1}{2ni}$$

assuming zero-filling only in F1 (not common in F2). For very large ni, very small couplings can be observed—often well below the linewidth of a high-resolution 1D experiment.

- A common rule-of-thumb is that $J_{HH} \ge dres 1/3$ will be observed in a COSY spectrum; this estimate is definitely a guideline only. $dres 1 \equiv digital \ resolution \ in \ F1$ defined above.
- The most efficient method of observing small J_{HH} is with long-range COSY, which involves this sequence and tau > 0 (typically tau = 50-200 ms).
- **cosy** loses sensitivity from the strong resolution enhancing sinebell (and sinebell-squared) apodization functions used to overcome the absolute value processing. Even so, minimum phase cycling (**nt**=4) usually gives sufficient sensitivity. GCOSY is even faster (**nt**=1, **nt**=2 is better).
- DQF-COSY (next section) removes all singlets, and gives much cleaner diagonals. Use this sequence after a COSY if crosspeaks close to the diagonal or on a strong singlet region are needed.
- **gcosy** is preferred if a PFG (e.g., hcx or bbswg) probe is installed. **nt=1** can be used with this sequence, reducing total acquisition time by 4.

2d Absolute Value COSY (relayh)



B. Critical Parameters

p1 = 90° for maximum sensitivity; 45° (typical) to reduce intermultiplet crosspeaks ni = usually set satisfactorily by cosy macro; sw1/2ni gives usable resolution; must

have ≤ 12 Hz, but want ~ 6 Hz if time allows for best results

nt = 4 is minimum phase cycle

sw = set using movesw macro (preceded by boxing selected area of spectrum)

sw1 = must equal sw for foldt macro (commonly used; recommended for fast-COSY)

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tau = set 50-150 ms typical for long -range COSY **d1** = best set $\ge 2 \times T_I$, but often run with **d1** ~ T_I

C. COSY 2d Acquisition

• for COSY, setup as in ${}^{1}H$ 1d and optimize **sw** with the **movesw** macro, then retake the data to ensure **sw** is set correctly

- move the parameters: assuming you ran the ${}^{1}H$ in exp1 and have nothing important in exp2, the command mp(2) jexp2 entered from exp1 will work
- from exp2 then use SETUP SEQUENCES COSY or enter the macro cosy or gcosy
- check that ni (sw1/2ni ≤ 12Hz/pt, ~6Hz/pt if time allows) d1 and np are correct (check time), then enter au to run cosy

D. Calibrations

- can use facility pulse width calibrations; COSY is very forgiving with pulse widths
- even so, performing a pw90 calibration is always recommended for all 2d experiments

E. 2d Data Workup and Plotting

- COSY data can be processed with the **do2d** macro, or use the **wft2d** command (see also **man('cosy')** or **man('gcosy')**
 - do2d applies symmetrization with the foldt macro
- general display and plot commands

dconi ; displays 2d's with color map (faster than contours)

plot projections before issuing the following command using menus (use the PLOT button)

pcon ; plots contours

pconpos ; plots phase sensitive contours with positive peaks

having 10 contours and neg peaks having 1 contour

pconneg ; opposite of pconpos

plot2dhr ; plots with high-resolution traces (must have 1d in

another experiment)